(12) UK Patent Application (19) GB (11) 2 027 223 A

(21) Application No 7925687
(22) Date of filing
24 Jul 1979
(23) Claims filed
24 Jul 1979
(30) Priority data
(31) 930458
(32) 2 Aug 1978
(33) United States of America
(US)
(43) Application published
13 Feb 1980
(51) INT CL[3] G02B 5/28
(52) Domestic classification
G2J 28
(56) Documents cited
GB 1497467
GB 1358738
(58) Field of search
G2J
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(54) Optical interference filter

(57) Optical interference filters comprising a silver layer adjacent a SiO_x layer, where x is from 1 to 2 and the layers have been vapour deposited, are provided with an intermediate layer of nickel, chromium or an alloy containing at least 90% of nickel and/or chromium, between the silver and SiO_x layers. The intermediate layer improves adhesion between the layers; it preferably has a thickness of 3 to 20 Å and is formed by vapour deposition.

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SPECIFICATION

Optical interference filters

This invention relates to optical interference filters and, more particularly, to such filters having adjacent silicon oxide and silver layers.

Infrared reflecting filters having adjacent refractory dielectric and silver coatings have recently been discovered. Such filters are described in our Application 7910526 Serial No 2017965 and are made by vapour depositing one or more three-layer coatings (such as a three-layer coating also being referred to as a period). Each period has a layer of silver sandwiched between two layers of a refractory dielectric oxide. Suitable refractory dielectric oxides include, for example, silicon oxides having the formula SiO_x, where x is from 1 to 2. The said application further discloses that palladium enhances the bond between the SiO_x and silver layers. However, palladium has the disadvantage of substantially reducing the visible transmission, even when used in films as thin as 5Å, and is also very expensive.

We have now found that thin coatings of certain metals (other than palladium) substantially improve the adhesion between adhacent vapour deposited layers of silver and SiO_x, where x is from 1 to 2. Suitable metals for this purpose are nickel, chromium and alloys having a nickel/chromium content of at least 90%, such as Inconel.

According to the present invention, therefore, there is provided an optical interference filter comprising a silver layer adjacent a SiO_x layer, where x has a value of 1 to 2 and the layers have been vapour deposited, in which an intermediate layer of nickel, chromium or an alloy having a nickel and/or chromium content of at least 90% is provided between the silver and SiO_x layers.

While the thickness of the intermediate layer is not particularly critical, a layer sufficient to provide enhanced adhesion between the adjacent layers without significantly reducing light transmission is preferred. A layer from 3 to 20 Å, more preferably 3 to 8 Å, thick is preferred for this reason, the layer preferably being formed by vapour deposition.

The interference filter according to the invention is preferably applied to a transparent plastics substrate, preferably formed of a polycarbonate, and a continuous abrasion-resistant transparent coating is applied over the interference filter thereby forming an infrared reflecting lens or welding faceplate.

Although it is the metal that is most sensitive to the coating rate and coating conditions when vapour deposited, chromium is the preferred metal for the intermediate layer if an abrasion-resistant coating is applied over the interference filter. Our Application 243/78 describes the preferred method and materials for providing such abrasion-resistant coatings. These coatings, as well as the prime coat, are applied as described in the said application; a specific example is given below. When an abrasion-resistant coating is used, chromium unexpectedly provides enhanced adhesion between the silver and SiO_x layers in the final product, even if adhesion was not significantly enhanced before the abrasion-resistant coating was applied. While the mechanism by which this occurs is not fully understood, the advantage of improved products is substantial.

In order that the invention may be more fully understood, the following examples are given by way of illustration only:

Example 1

Using conventional vapour deposition apparatus and procedures, four plano polycarbonate lenses were coated on one side at room temperature with an interference filter having adjacent SiO/Ag layers. Initial pump down for coating was to 1×10^{-5} Torr with the individual layer thicknesses and O_2 partial pressures indicated below.

LAYER NO.	LAYER MATERIAL	O_2	LAYER THICKNESS
1	SiO	3×10^{-5}	550Å
2	Ni	–	6Å
3	Ag	–	100Å
4	SiO	2×10^{-5}	550Å
5	SiO	2×10^{-5}	550Å
6	Ni	–	6Å
7	Ag	–	100Å
8	SiO	2×10^{-5}	550Å

Example 2

Two other runs having two lenses coated in each were conducted following the same procedure, but using an initial pump down of 9×10^{-6} and 8×10^{-6}, respectively. The silver layers were deposited more slowly in the run having the higher pressure and these slowly deposited coatings failed a pressure-sensitive tape test for adherence. All other lenses of Examples 1 and 2 passed the adhesion tape test.

The adhesion tape test was carried out as follows:

A piece of 1/2" pressure-sensitive regenerated cellulose tape (Scotch Brand manufactured by 3M Company) was firmly pressed on the surface for a distance of about 1". While holding the lens in one hand, the free end of the tape was jerked in a direction perpendicular to the lens surface. No separation of coating(s) is required for a "passed" adhesion report. Although, separation between layers or between the first layer and the substrate usually begins at the edge (where the coating(s) stop), separation occurring at any location results in a "failed" report.

When a continuous abrasion-resistant coating is provided as the outer coating on the lens to protect the filter coatings, the lens surface with the filter coatings is cross-hatched with cuts extending to the substrate and approximately 3 to 5 mm. apart before applying the tape. One end of the pressure-sensitive tape is then pressed on the cross-hatched coating(s) and the other end is then jerked in a direction perpendicular to the surface. The multiple edges offer a greater opportunity for adhesion failure and this test is considerably more severe than the normal adhesion test.

The speed with which vapour deposition takes place is a function of the gun power, type of crucible, gun focus, and the distance from the material source to the workpiece. In a chamber having a source to workpiece distance of 48 cm., a deposition rate of about 1Å/Sec. is required for "fast" depositions of nickel, chromium and Inconel and about 10 to 15Å/Sec. for silver. The rate of silver deposition was about 2Å/Sec. for the "slow" run of Example 2. While the deposition rate is not critical to the adhesion of the SiO_x layer, the latter are usually deposited at a rate of 15Å/Sec.

Example 3

Following the procedure of Example 1, two polycarbonate lenses were coated with the interference filter. One lens was coated without adhesion-promoting coatings and the other with 6Å of chromium as the adhesion-promoting coating material. The lens without the chromium failed the tape adhesion test, while the lens with the chromium coatings passed the test. When using chromium, care must be taken to allow the chromium source to cool nearly to room temperature before opening the chamber in order to prevent the formation of oxides on the source. Such oxides act as contaminants in subsequent coating operations. Chromium can be evaporated by resistance heating methods, but an electron beam gun is the preferred method.

Example 4

Using the procedure of Example 1, two runs were made coating two polycarbonate lenses in each run with Inconel as the adhesion-promoting coating instead of nickel. The first run had coatings of 6Å of Inconel deposited in about 5 seconds each and the second run had coatings of the same thickness deposited in about 30 seconds each. Both lenses of the first run passed the tape adhesion test, while both lenses of the second run failed the test.

Example 5

A number of polycarbonate lenses were coated using the procedure of Example 1, except chromium was used instead of nickel. After applying the interference filter, the lenses were divided into two groups when it was discovered that adhesion between the chromium and silver was poor. In fact, all lenses of one group failed the tape adhesion test. The poor adhesion was probably due to either to not cooling the chromium source before opening the chamber after a prior run or depositing the chromium at too slow a rate. The other group of polycarbonate lenses having the interference filter coatings were immersed at room temperature in a tie-coat solution containing 10% gamma-amino propyl triethoxysilane, 85% ethyl alcohol and 5% water for one minute. The primed lenses were then removed from the silane solution, rinsed in water and dried.

The primed lenses were then coated with an abrasion-resistant coating sold by Dow-Corning under the designation Q9-6312 and understood to be covered by U.S. Patent 3,986,997. The Q9-6312 material was filtered upon receipt, placed in a container and the lenses were immersed and then withdrawn at a controlled rate of 7.5 inches per minute. The lenses were then cured in an air-circulating oven for eight hours at 250°F. All the lenses in the group having the abrasion-resistant coating passed the more severe cross-hatched tape adhesion test in spite of the adherence problem existing before applying the abrasion-resistant coating.

CLAIMS

1. An optical interference filter comprising a silver layer adjacent to SiO_x layer, where x has a

value of 1 to 2 and the layers have been vapour deposited, in which an intermediate layer of nickel, chromium or an alloy having a nickel and/or chromium content of at least 90% is provided between the silver and SiO_x layers.

2. An interference filter according to claim 1, in which the intermediate layer has a thickness of from 3 to 20 Å.

3. An interference filter according to claim 1 or 2, in which the intermediate layer is formed of chromium and has a thickness of 4 to 6 Å.

4. An infrared reflecting lens or welding faceplate, which comprises:
(a) a polycarbonate plastics substrate,
(b) an interference filter bonded to a surface of the substrate (a) and comprising
(i) a layer of SiO_x, where x is from 1 to 2,
(ii) a first intermediate layer of chromium, nickel or an alloy having a nickel and/or chromium content of at least 90%,
(iii) a layer of silver,
(iv) a second intermediate layer as specified for (ii),
(v) a layer of SiO_x,
said layers (i) to (v) being vapour deposited, and
(c) a continuous abrasion-resistant transparent coating over the interference filter.

5. A lens or welding faceplate according to claim 4, in which layers (b) (ii) and (iv) are formed of chromium and have a thickness of 4 to 6 Å.

Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon) Ltd.—1980.
Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.